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ABSTRACT

This report presents the results of an extensive literature survey undertaken to establish optimum sampling, sample handling and long-term storage techniques for a wide variety of environmental samples to retain sample integrity. The components of interest are trace elements, organics, pesticides, radionuclides and microbiologicals. A bibliography is provided for reference. (CS)

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A Survey of Current Literature on Sampling, Sample Handling, and Long Term Storage for Environmental Materials

SE 023 750

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E. J. Maienthal and D. A. Becker

This article is the result of an extensive literature survey to establish optimum sampling, sample handling and long term storage techniques for a wide variety of environmental samples to retain sample integrity. The components of interest in these samples are trace elements, organics, pesticides, radionuclides or microbiologicals. This survey was done both manually and by use of various bibliographical retrieval services. Also the advice and opinions of workers in various aspects of the fields was obtained.

Key words: Environmental samples; long-term storage; microbiologicals; organics; pesticides; sample handling; sampling; chemical analysis; radionuclides; trace elements.

1. Introduction

In order to aid in the development of criteria for the possible establishment of a National Environmental Specimen Bank for the Environmental Protection Agency, a large portion of the recent literature concerning sampling and storage of environmental specimens has been examined. This has been done both manually and by use of bibliographical retrieval services such as Medline, Chemcon, Biosis, Cain, Defense Documentation Center and others. Also, the advice and opinion of workers in various aspects of the field has been obtained. A summary of the results of this survey is found below, separated into the various areas of concern.

2. Trace Elements

For trace elements there is an abundance of reports on sampling and storage (which should also apply to radionuclides); however, many of them are contradictory and should be further resolved by careful experimental work. Much of the published analytical data apparently is inaccurate because of such problems as gross sampling contamination or subsequent procedural contamination and failure to make proper blank corrections. Richards states that some oceanographers have permitted the perpetuation of the

notion that the concentrations of trace elements in the sea are well known, when, in fact, they are not (1).[1]

Patterson and Settle (2) report that the great mass of published lead data in plants, animal tissues and water is in error because of gross positive errors, and that the relatively large blanks usually present with lead concentrations less than a few μg/g often makes the value obtained meaningless. Many trace element analysts, particularly in the field of oceanography and marine biology, believe that much of the previously published work is unreliable as a result of sample contamination. The values being reported are progressively lower as techniques are being improved. Hume reports that if a synthetic sea water were prepared from the purest reagent chemicals available, it would still be higher in many trace elements than natural sea water (3). Whitnack also has evidence to show that the reagents used are more contaminated than sea water (4). Speecke, et al. state that many chances exist for a biological material to be contaminated before it is analyzed (5); but few authors give the impression of the awareness of this and meaningless phrases are used, such as "metal-free" containers, "chemically clean" glass, etc., with no evidence to back it up. Berman states that one must never assume anything is acceptably free from trace metal contaminants until it has been tested (6).

It is felt that the materials, techniques, and expertise exist to provide viable long-term stored samples for most trace elements in most matrices; however, few in the field are using these techniques, partially because as Boutwell says, "...validity is an expensive commodity" (7).

The first consideration is the choice of the container and sampler composition and the method of cleaning and sampling. Murphy, Robertson, Thiers, Patterson, Tölg, and many others show results which indicate that rubber, neoprene, vycor, polyvinyl chloride, polystyrene, glass, polypropylene, linear polyethylene, platinum, etc., will introduce contamination in sampling and storage (8, 9, 10, 10a, 2, 11). Patterson recommends first, FEP Teflon, then ultrapure quartz, conventional polyethylene or TFE Teflon containers. All cleaning and sample treatment should be done in laminar flow hoods or a clean room. He recommends cleaning with hot concentrated HNO_3 for three days, rinsing with high purity distilled water, followed by hot dilute 0.05 percent HNO_3 (both water and acid, prepared as described by Kuehner, et al. (12)) for one day, rinsing and heating with 0.05 percent HNO_3 five days, rinsing, then storing filled with 0.05 percent HNO_3, wrapped in cleaned polyethylene until ready for use. (The two dilute HNO_3 leachings have

[1] Figures in parentheses indicate the literature references on page 16.

not been found necessary by some other workers). When ready for use, the containers can be thoroughly rinsed and dried in laminar flow hoods. Cleaned plastic gloves are worn in all phases of cleaning, sampling, etc. (2). Berman found that even after thorough cleaning and scrubbing of fingers, 0.1 to 0.4 µg of lead could still be washed off. Washings from a chain smoker give results of 0.3-4 µg of lead (6).

Karin, et al. report a three-day leach of polyethylene in either 8 or 16 M HNO_3 was necessary to remove certain trace metal contaminants (13).

Sampling implements Patterson recommends are either Teflon or Teflon-encased, except for frozen tissue sampling where a series of HNO_3-acid cleaned stainless steel blades are used with very elaborate sampling procedures to remove areas contaminated by the blade (2). All of these type operations should be done in laminar flow hoods or clean room conditions.

Deionized water which has not been followed by distillation should not be used in any stages of the cleaning, sampling, or analysis as organic breakdown products may be formed, complexing some of the trace elements (8).

Numerous types of water samplers have been devised. Segar, et al. have described water sampling with Niskin bottles with rubber coated springs, Teflon coated coil springs and a new design Niskin bottle without internal closures. All gave trace metal contamination except the latter (14). Since Teflon is rather porous, apparently some metal diffusion through the spring coating must have occurred.

Harrison, et al. have designed a Teflon cylindrical sampler with a mechanism for opening both ends after submersion to the desired depth to avoid contamination from the water surface (15). It is attached to a metal frame and rudder which have a baked-on Teflon coating. It is also adapted so that the sample may be filtered immediately in an attached container holding a precleaned polyethylene bag in which the sample can be immediately sealed and frozen in liquid nitrogen. If the water sample is to be filtered, Morrison and Pierce, and many others suggest that it is best to do it immediately (16). The filter must be thoroughly precleaned, rinsed, and stored in cleaned polyethylene bags.

The sample chamber used by Patterson (2) consists of accordion pleated Teflon tubing, the entry port being protected by a bath of ultra pure water (prepared as already mentioned). At the deep water sampling depth desired, a

trigger retracts the water bath shroud and ruptures the end diaphragm which contains the pure water. The water sampler is lowered continuously so that it is continually dropping into virgin water. After a short interval to allow the bath water to be washed away, a second trigger expands the sample accordion bag and seals the entry port.

The storage of aqueous samples presents an even greater challenge as most samples start undergoing changes the instant they are sampled. Pre-aging the sampler and sample container with some of the same sample would be desirable whenever possible. Amore states that losses as high as 50 percent can occur during one hour of storage (17). An EPA manual on methods of water analysis says that complete and unequivocal preservation of samples is a practical impossibility, that complete stability can never be obtained, and that preservation techniques only retard the chemical and biological changes that continue after the sample is taken (18). The methods of preservation are intended to retard biological action, retard hydrolysis of chemical compounds and reduce the volatility of the components. Their recommended methods include pH control, chemical addition, refrigeration, and freezing.

Although there is much in the literature on relatively short term storage of different aqueous (non-frozen) solutions under varying conditions, there are many disagreements and most of the results do not look favorable for long term storage. A USGS manual for water analysis says that the shorter the time that elapses between the collection of a sample and its analysis, the more reliable will be the results (19).

Pettis and Phillip give an excellent review of the literature on trace metal analysis in sea water. They discuss sampling and cleaning procedures, sample pretreatment, standard reference materials, and analytical method of determination of the trace metals (19a).

Robertson found that sea water adjusted to pH 8 stored in polyethylene resulted in a 90 percent indium loss in 20 days and a 90 percent loss of iron in 55 days (20). Hummel found that 75 percent of the gold in sea water was lost after three weeks in polyethylene (21). King, et al. found that less than 3 percent of the cadmium was lost to polyethylene at pH's of 3 to 10 after two weeks storage (22). West, et al. (23) found more silver adsorption on glass at pH 4 than at pH 7, a significant decrease occurring at pH 7, and a rise at pH 8, and they also state that pyrex showed more erratic adsorption patterns than polyethylene or silicone-coated containers. Struempler (24) states that acidi-

fication with nitric acid to pH 2 prevents adsorption of silver, lead, cadmium, and zinc on pyrex, and silver on polyethylene. Dyck (25) reports lack of confirmation with the work of West, et al. with silver, and states there is a direct increase in silver adsorbed on glass with increase in pH. He also states that for periods over several months, plastic adsorbed more silver than glass. Lai and Weiss (26) found no silver loss when sea water was stored in polyethylene and acidified to a pH of 3.5 to 4.0 with acetic acid. King, et al. (22) found losses as high as 75 percent for cadmium when stored in glass at pH 9. Eichholz, et al. (27) compared adsorption of a number of elements on pyrex and polyethylene and state that pyrex is preferable to polyethylene; however, they found less contamination for cesium, ruthenium, and zirconium when using polyethylene. Smith (28) studied stability of a number of ions including cadmium, antimony, tin, and lithium, and states that of the elements studied only lithium was stable over the pH range of one to 11. He therefore recommends acidification to pH one. In another report (29), he states that freezing the liquid samples as soon as they are collected is an excellent solution for the adorption problem. The losses may be due to adsorption or also to precipitation or particulate formation. Salman also states that freezing can be used to preserve the water samples at the collection site (29a).

Rattonetti examined the stability of a large number of trace metals in a variety of water matrices stored in polyethylene at differing pH's and concluded that loss to container walls is insignificant compared to losses to the particles present in natural aqueous systems (30).

Moody, et al. prepared two mercury-in-water Standard Reference Materials at the 1 ppm and 1 ppb level which have been stable for over a year in both glass and polyethylene (31). This was achieved by the addition of 1 µg/g and 10 ng/g of Au^{+3}, respectively, and 0.5 N nitric acid. Lo and Wai verified this for shorter term storage with 0.2 µg/g Au^{+3} and nitric acid at pH 0.5 (32), but were unable to confirm Feldman's stabilization with potassium dichromate (33) or the report of Issaq and Zielinski with hydrogen peroxide (34). Avotins and Jenne state that the biological effects have been overlooked in many mercury in water investigations, and as a result of unpredictable growth of bacterial and yeast populations, with production of metabolites, mercury may either vaporize, bind to the walls of the vessel or be stabilized in solution (35). Huey, et al. have reported that cadmium can be volatilized from its inorganic salts by a microorganism through conversion to a volatile organic compound (36). The volatilization is stimulated by vitamin B_{12}. Methylmercury formation by this organism is

also stimulated by B_{12}, the absence of which causes the organism to form metallic mercury from inorganic mercuric salts. In samples where this type of reaction occurs, freeze-drying is not advisable as a method of sample preservation. For long term preservation for trace-element analysis, freezing and possibly freeze-drying for most elements (probably followed by radiosterilization), would seem to be the most likely alternatives. Morrison and Pierce state that freezing may be a suitable preservation technique for trace elements but has not been adequately tested to date (16). Allen, et al. recommend immediate freezing at -10 to -15°C to prevent microbiological changes in soluble mineral and silica concentrations (37); however, for long term storage, immediate freezing in liquid nitrogen as recommended by Harrison, et al. (15) and others (29), followed by freeze-drying for most trace elements (and radiosterilization) or storage at -70° to -80°C would seem preferable. Low temperature (oxygen plasma) ashing and dry ashing are also possibilities in some cases.

Harrison, et al. (38) and Filby, et al. (38a), have reported that radioisotope studies of the volatile elements such as arsenic, antimony, selenium, bromide, and mercury have shown no significant losses in water samples which have been freeze-dried.

Heron studied the determination of phosphate in lake water before and after freezing (39). It was expected that rapid freezing would cause cell rupture resulting in higher phosphate values, but this did not occur. Varying phosphate values were found whenever growth of bacteria was occurring. This was prevented by pre-cleaning the sample bottle with a solution which is 5 percent in iodine and 8 percent in potassium iodide and immediate freezing of the water sample.

Philbert found that in freezing lake water samples soluble reactive silica and phosphorus concentrations were decreased in the thawed samples (40). A decrease in total alkalinity and dissolved chloride was also observed. Inconsistent changes were observed for ammonia and the various forms of nitrogen.

A USGS manual on methods of water sampling recommends that water samples for inorganic analysis should not be frozen (19); however, there is sufficient reason to expect that if the process is performed properly, freezing is acceptable for most trace elements. The samples should be subsampled before freezing, because once thawed, they should not be refrozen. The entire subsample should then be taken for analysis. They should be frozen in one of the container materials already discussed, under a gas such as nitrogen or

argon to prevent sample oxidation. They should be sealed in at least 2 and possibly 3 [as Patterson recommends (2)] series of plastic bags. Since most plastics are porous (41), they should then be placed in a tightly sealed glass container containing nitrogen or argon with minimum void space, followed by storage in the dark at -70°C. Bothner and Robertson (42) have reported that sea water samples stored in polyethylene containers have picked up mercury from being stored in a room contaminated with metallic mercury. This has been verified in a closed chamber with pools of clean mercury surrounding a mercury solution in Teflon and polyethylene bottles, but has not as yet been verified in an ordinary laboratory atmosphere where spilled mercury would probably be covered with dust, thus effectively diminishing its vapor pressure (43).

When the frozen water sample is used, the whole sample should be used because of possible selective ion incorporation in the ice (44). The walls of the inner container will probably have to be washed with acid to remove any hydrolyzed or adsorbed material.

The possibility of losing organic or inorganic mercury during freeze-drying of biological materials was investigated by LaFleur, as Pillay, et al. had published data indicating losses (45). LaFleur found no losses for inorganic or naturally bound methyl- or phenylmercury in tissue and blood; however, for aqueous solutions, losses of up to 90 percent could occur for organic and up to 10 percent for metallic mercury (46).

3. Biological-Tissue and Fluids

For tissue and biological fluid sampling, the sampling device presents considerably more difficulties. The use of a laser beam for cutting bone by Hislop and Parker (47) offers many interesting possibilities. Some loss of trace elements on the surface may occur but would be negligible with regard to the entire sample. A quartz or glass knife should also be suitable for many kinds of tissue. Montgomery, et al. used a glass knife to cut fish in small pieces for the determination of iron, zinc, lead, cadmium, copper, and manganese (48). A problem here is the chipping of the cutting edge; weighing the knife before and after use may indicate if this difficulty arises.

Most workers use stainless steel implements. However, this is frought with dangers of contamination for many trace elements even when done as carefully as described by Patterson earlier (2). Versieck, et al. report on the contamination introduced during needle biopsies of liver (49). They state that steel surgical blades lead to somewhat less contamination,

but are not suitable for some trace elements such as chromium and nickel. The needle biopsies resulted in contaminations of as much as 1.7 ppm of copper, 0.64 ppm of manganese, 11 ppm of chromium, 12 ppm of nickel, 20 ppm of iron, 0.24 ppm of cobalt, 0.012 ppm of silver, 0.46 ppm of tin, 0.069 ppm of antimony and 1.2 ppm of tantalum. Speecke, et al. have reported on the sampling and storage of biological materials for contamination by chromium, manganese, nickel and cobalt by drawing 4 series of 20 ml portions of blood using disposable needles (5). For manganese, the first 20 ml showed contamination of 0.2 ppb, the fourth, 0.02 ppb; for chromium, the first 85 ppb, the fourth 15 ppb; for nickel, the first 71 ppb, the fourth 12 ppb; for cobalt the first 0.9 ppb, and the fourth 0.2 ppb. They also compared contamination introduced in another series of liver samples using Meneghini biopsy needles and surgical blades. For the needles, they found contaminations of as much as 600 ppb of manganese, 9000 ppb of chromium, 12,000 ppb of nickel and 230 ppb of cobalt; for the surgical blades, 3 ppb of manganese, 15 ppb of chromium, 60 ppb of nickel, and 1 ppb of cobalt. They discuss the possibility of using laser beams on hard and soft tissues and platinum-rhodium alloy needles; however, it is preferable that the platinum needles have Kel-F hubs to avoid contamination. For storage, Speecke, et al. recommend immediate, rapid freeze-drying, but point out that some volatile materials may be lost. All the work should be done in a clean-room type laboratory with no exposed metal parts which might cause contamination.

Fisher, et al. (50) also reported that serum samples should be quickly frozen with as little air space as possible (as described earlier, the air should be displaced with nitrogen or argon). They also checked storage at room temperature, 8°C and -15°C. No differences for calcium, magnesium, copper, zinc, sodium, and potassium were noticed up to 16 days. Essentially no changes were observed in the refrigerated and frozen samples up to 50 days, but changes did occur in the samples stored at room temperature. Longer term storage would probably also result in changes in the refrigerated samples. Some microorganisms can grow in a temperature as low as -6°C (51).

In a discussion of sampling for clinical chemistry, Ibbott recommends separating the serum from the clot as soon as possible to avoid contamination from cell leakage (52). He also states that the majority of the serum components are stable indefinitely in dry ice (about -70°C), and that the samples exhibit concentration gradients due to freezing and must be thoroughly mixed after thawing. Omang and Vellar also point out the concentration gradients obtained after freezing and thawing serum, sweat, and urine. They found top-bottom differences of thawed samples of up to one hundred (53).

4. Museum Specimens

The futility of trace element analysis of museum type specimens stored in preservatives has been pointed out by a number of authors. Bowen and Sutton in analysis of marine sponges found that nickel accumulation in the preservative occurs quite frequently in these types of samples (54). Gibbs, et al. investigated the effects of time and preservatives on museum fish specimens and found no evidence to support the theory that preserved museum specimens can provide reliable estimates of heavy metal concentrations (55). They tested many types of preservatives such as ethanol, formalin, isopropyl alcohol, etc., and found interaction with the specimens in all cases. These solvents may either leach trace metals from the specimen or contaminate the specimens by heavy metals contained in the preservatives or container. In many cases, metal identification tags are placed in with the preservative, which contribute even further to the contamination of the sample. In some instances, the trace metal content increased over the years and in other cases, decreased from leaching even in a short period of time, such as a month.

A possible exception for the museum type specimens are those which have been stored in relatively clean, dry areas not subject to leaching or contamination. Cockburn, et al. (56) describe the autopsy of an Egyptian mummy, Pum II, which included the analysis of some trace elements in bone by R. G. Smith (57). He found 0.6 ppm of lead and 0.43 ppm of mercury. The lead content of modern bone averages 6.55 to 18 ppm (58). Assuming no leaching has occurred, it would appear, on the basis of limited sampling, that man's environment has contributed considerably to his lead body burden. The mercury level, however, is relatively unchanged, that of modern bone averaging about 0.45 ppm (59).

5. Crustal and Botanical Materials

The sampling and storage of soils, rocks, minerals, sediments, and plants does not present quite as many problems as the matrices already discussed, but more precautions should be taken then are generally observed. Morrison and Pierce (16) state that the use of a spade to sample soil is preferable to a soil auger and that dry samples can be collected in a clean cloth bag, but this procedure would certainly lead to contamination for some trace metals. Clean Teflon encased tools as recommended by Patterson and Settle (2) should be used except for most plants which can be picked with clean plastic gloves. It appears that soils and sediments with any significant water content (especially sediment samples) should be frozen in such a way that no water loss can occur, and stored as recommended for water samples.

There are many papers in the literature which indicate that soils and sediments undergo changes in structure and chemical state even when dried at room temperature. This should not have a great effect on the total trace element content in most cases, but if speciation, organic extractable trace elements, etc., are of interest, any form of drying may invalidate the sample. Attoe (60) reports that potassium may be fixed in a nonexchangeable form when a potassium-fertilized soil is air-dried. Air drying of unfertilized soils resulted in a 4-90 percent increase in exchangeable potassium when the soils are remoistened. Schalsha, et al. (61) state that air drying produces significant irreversible changes in volcanic ash soils. For instance, soil samples with a clay-type texture in the field, change to a sandy texture with air drying. Air drying also reportedly markedly affects cation exchange capacity, soluble phosphorus and iron, and decreases the pH slightly. Air drying decreased the total exchangeable and acid soluble iron, but increased the chelatable iron extracted by salicylate. The mechanical and chemical analysis of volcanic ash soils more accurately indicate field conditions when samples contain the original moisture at field capacity.

Barrow (62) found when soils were dried, inorganic sulfate immediately increased (probably as a result of decomposing organic sulfates in the soil becoming immediately available to the plants). Even when two different soils are dried at the same temperature, the relative availability of the sulfur may be no indication of the relative availability when they were fresh.

Harpstead and Brage (63) reported that the drying and storage of soils leads to a pronounced increase in their nitrifying ability because of the changes in the relative numbers of various microorganisms in the soil. Birch found that when remoistening dried soil, the first rapid decomposition slows down and this pattern is repeated during successive dryings and wettings (64). The magnitude of the decomposition depends on the amount of carbon in the soil and on the drying conditions, air drying being less effective than oven drying. Vacuum drying and oven drying gave the same moisture loss results, but oven drying gave a much greater amount of decomposition on rewetting.

Birch (65,66) also states that the longer a soil is kept air dried, the greater the amount of water-soluble and organic material that can be extracted, even though it does not lose additional moisture, and also the greater the amounts of carbon and nitrogen are mineralized on remoistening. He also finds greater effects if the soil is dried at 100°C, possibly because of increased gel porosity and

surface area, and possibly because of increased microbiological activity occurring during the remoistening of the dried soil.

Nevo and Hagin (67) state that the changes occurring after three months of air drying storage was independent of microorganisms. The major factor is the change in the physical structure of the organic fraction. They found a good correlation between the nitrification rate and the surface area of particles of an organic soil.

Hesse (68) states that over drying a soil, despite its reproducibility, should not be recommended, because of the profound changes caused. Also he says that storing a soil in a moist state has the effect of incubating it, but without temperature or moisture control, resulting in a build-up of carbon dioxide at the expense of oxygen. As such treatment results in many complicated reactions, it is most undesirable to keep a soil in a moist state for any length of time for the purpose of analysis. He also reports on investigations of J. M. Coleman (private communication) that moist soil samples stored in plastic containers can result in fundamental changes in clay minerals. It is thought that an organic complex passes from plastic into the clay mineral. All these references seem to point out that drying or freeze-drying may result in irreversible changes which will affect also the complexation state of the trace metals and that freezing at -70°C to -80°C as recommended earlier should be the method of storage.

Plant sampling can probably be done by picking with plastic gloves and storing by freezing in containers as already mentioned, with care to avoid moisture loss.

Arkeley, et al. (69) state that trace elements such as carried by peat dust deposited on plants are easily washed off (high purity distilled water should be used) but those deposited by sprays are not, because of partial absorption in the leaf. Lagerwerff (70) found increases in absorption of cadmium, zinc, and lead on leaf surfaces probably enhanced by drying.

Work by Koeppe and Miller (71) showed a much higher uptake of lead by maize roots than in the stems or leaves. Washing with distilled water removed little lead, but washing with EDTA solution removed about 90 percent of the lead, indicating the lead is largely retained on the exterior surface of the roots.

For sampling of air particulates, Patterson and Settle (2) recommend cleaning Millipore or Nucleopore filters by soaking in cold 6N HCl two days, rinsing on a cleaned

polyethylene Buchner funnel with high purity distilled water, soaking two days at 55°C with 1 percent NH_4F (prepared by neutralizing high purity NH_4OH with high purity HF) followed by rinsing with high purity water. These operations, of course, are carried out in a clean room atmosphere or laminar flow hoods. The filters are then stored in cleaned polyethylene bags or boxes. The lead blank on these filters was found to be less than 1 ng/47 mm filter.

6. Organics and Pesticides

With the exception of the use of plastic gloves for sampling to avoid contamination from body oils (72), storage containers and implements for trace organics and pesticides definitely must not be plastic of any kind, with the possible exception of Teflon, as plastic is known to both introduce interferences and sorb pesticides (and organics) (73,73a). Many examples are given in the literature which show that additives such as plasticizers, organometallic or other stabilizer antioxidants, colorants or other components are leached from the plastic and contaminate the sample (74).

Some polyvinyl chloride tubings were shown to release a constituent to some systems containing alcohol, propylene glycol or polyethylene glycols (75). Gibbs found that asbestos fiber was highly contaminated by 3,3'-5,5'-tetra-tertiary butyl diphenoquinone after storage in polyethylene bags (76). Lipids in soil samples stored in standard plastic lined canvas bags were found to take up phthalate esters and other contaminants from the plastic (77).

Most workers in the field recommend storage in glass containers with Teflon or aluminum foil lined caps (72,73, 73a,78); however, it has been reported that Teflon sheet and aluminum foil have been found to contain up to 400 and 300 ppb, respectively, of di-2 ethylbutyl phthalate (79,80).

Hertz, et al. (81) recommended cleaning the glassware with soap and water, then in concentrated H_2SO_4 at 100°C for 30 min and finally rinsing with specially prepared distilled water made by redistilling the house distilled water over $KMnO_4$-KOH. The distillate is then passed through an XAD-2 column, and the water is redistilled to remove any particulates from the XAD-2 resin. Finally, the bottles are then rinsed with methanol and triple-distilled pentane, and filled with nitrogen from a liquid nitrogen source and sealed.

Others recommend wrapping the cleaned glassware in aluminum foil and heating at 625°C for four hours (82). The maintenance of high quality distilled water can be a problem as some microorganisms can grow rapidly in distilled water

and some chemical reagents (82,83,84). It is reported by Hamilton and Myoda (82) that the amino acids, proteins and bacteria often found in some laboratory reagent solutions and distilled water, are probably airborne and enter the outlet of the stills or deionizing systems where they multiply. A method of catalytic pyrodistillation has been reported to remove organic impurities not removed by ordinary or oxidative distillation because of the steam volatility of the compounds or their derivatives (85).

It has been recommended in sampling that "an analyst or person directly concerned with the particular study should collect the samples. Inexperienced personnel should never be allowed to collect the samples unless they are very closely supervised" (78). This, of course, is true to all types of environmental sampling.

When sampling marine organisms and sediments for organics or pesticides, most workers recommend freezing immediately in dry ice or liquid nitrogen (72,81) and final storage at about -70° to -80°C (86) in the dark. Breakdown of pp'-DDT to pp'-TDE in Bengalese finch liver, and breakdown due to other biological processes have been reported at home freezer storage conditions (approximately -14°C) (87,88).

Bristol reported in a study of pesticide residues in potatoes that metabolically incorporated 2,4-D untreated potato samples stored whole at 4°C decreased over a period of 15 months, while those of 2,4-DCP remained constant. Recoveries of 2,4-D from frozen samples were constant over a 15-month period, but those of 2,4-DCP decreased slowly from 88 to 47 percent. The 2,4-DCP samples stored in plastic bags gave a characteristic odor, indicating the losses were due to volatilization from the frozen samples (89).

It is reported by the Federal Working Group on Pest Management that increased knowledge of sample contact with various kinds of synthetic wraps and containers demonstrates the necessity for glass and perhaps aluminum foil to preserve the integrity of wet samples. Immediate freezing and maintenance of the frozen sample until analysis is the best way to protect samples and prevent degradation and loss of pesticide residues (73). (This also is undoubtedly true for all organic components.) They also state that pesticides can migrate to the walls of a container and be adsorbed; hence, even with a glass container, after the sample is poured out, the walls should be rinsed with the solvent in case the extraction is not made in the container itself (this should also apply to any organics).

There is evidence in the literature that samples to be analyzed for organics or pesticides cannot be dried or

freeze-dried without danger of some loss. One study showed 79 percent loss of lindane, 37 percent for dieldrin, 57 percent for p,p'-DDT and 31 percent in o,p'-DDT-DDD on whole eggs and 50 percent for lindane in egg yolk when samples were frozen at -23°C, freeze-dried for 24 hr, and transferred and stored in sealed glass vials at 4°C, so that there was no volatility loss in storage (90).

Morris found that preservation of zooplankton in formalin and methanol resulted in hydrolysis of the animals' lipid and degradation of polyunsaturated fatty acids (90a). He found that the samples were stable up to nine months if stored deep frozen under nitrogen.

Smith reports that changes in nonstructural carbohydrate concentrations occur during the storage of either heat or freeze-dried tissues and concluded that no preservation method is as good as the immediate analysis of fresh tissue; however, he did not investigate straight freezing (91). Other workers found losses in higher fatty acids under either oven or freeze-drying conditions after storage for nine months (92).

Dessicants for tissue preservation are used by some workers who are unable to freeze their samples. The samples are chilled, homogenized, and blended with a combination of sodium sulfate and powdered silica. It is stated that the resulting mixture is a dry, free-flowing powder with the pesticide residues are stable for 15 days or more at room temperature (73).

7. Microbiologicals

Microbiologicals or cellular organisms consist of many different types such as algae, protozoa, fungi (molds or yeasts), bacteria, submicroscopic viruses and other (microscopic nematodes, some insects and some crustaceans), necessitating a wide variety of different sampling and storage conditions. Most preservation has been done through culturing and subculturing. With care, these have been maintained for 5-8 years (93). The sampling implements and containers should obviously be sterile and glass is preferred to plastic, since bacteria tends to grow on plastic surfaces. Since all known life forms require water in the liquid state, this automatically limits the temperature range for microorganisms. Both bacteria and viruses can be freeze-dried to maintain culture collections and to preserve them for use as vaccines. Insects have been supercooled to -30°C without apparent damage; however, they die if ice crystals are formed. Mouse embryos have survived deep freezing to -196°C. Freezing is accompanied by the removal of water, so the cell is subject to damage by both freezing and drying.

Mechanical injury is caused by ice crystals and the removal of the water causes an increase in dissolved substances. Biochemically debilitated cells may show a reduction or complete loss of some enzymes, and ice-damaged cells may have leaky membranes or an altered structure. The damage from freezing, drying, and thawing can range from essentially none to 100 percent, depending on the specific organism and conditions. Spores are resistant to both cold and drying. Rapid freezing is reported to be usually better with bacteria, whereas slow freezing is better for animal cell survival. It has been stated that rapid thawing gives better survival than slow thawing (51). Fleischer and Kervina report in studies on long-term preservation of liver for subcellular fractionation that rapid freezing and thawing minimizes the time in which degradation can occur (94). Repeated freezing and thawing is more harmful. Freeze-dried bacteria are better kept at refrigerator temperature than at room temperature (51).

McPeak and Camp (94a) have reported on work of Valeri, et al. (95), Meryman and Hornblower (96), and Gibson, et al. (97). Their studies on storage of red blood cells show that if the samples are stored at a higher temperature than -60°C, they deteriorate within a few weeks. If glycerol is used as a cryoprotective agent and the cells are frozen rapidly in liquid nitrogen and stored at -80°C, the cells are reported to be stable for over 10 years. Fluctuations in storage temperatures of not more than 10°C above or below -80°C are reported to have no adverse effect. Farrant, et al. (98) report that improved recovery of frozen cells can be obtained by interrupting rapid cooling with a timed exposure to a single subzero temperature.

There is a vast amount of additional information in the literature on the subjects of sampling, handling, and storage for microbiologicals, blood, and other biological samples. Since there are so many different types of species and the related optimum handling appropriate to each specie, it is difficult to summarize; however, a number of additional references are given below to indicate the type of problems that are encountered as well as some additional references concerning other subjects discussed above.

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17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)* Environmental samples; Long-term storage; Microbiologicals; Organics; Pesticides; Sample handling; Sampling; Chemical analysis; Radionuclides; Trace Elements

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